

FORM PTO-1390 R9REV 5-93)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C.371				P1998J130
INTERNATIONAL APPLICATION NO. PCT/GB99/00064		INTERNATIONAL FILING DATE 08 January 1999		U.S. APPLICATION NO (If known, see 37 CFR 1.5) 09 / 600011
TITLE OF INVENTION		GYPSUM PRODUCT		
APPLICANT(S) FOR DO/EO/US		BOELEE, Scotia		
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:				
1.	<input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.			
2.	<input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.			
3.	<input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l).			
4.	<input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.			
5.	<input type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)) a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau) b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US)			
6.	<input type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)).			
7.	<input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau). b. <input type="checkbox"/> have been transmitted by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input checked="" type="checkbox"/> have not been made and will not be made.			
8.	<input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).			
9.	<input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).			
10.	<input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).			
Items 11. to 16. below concern other document(s) or information included:				
11.	<input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.			
12.	<input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.			
13.	<input type="checkbox"/> A <b>FIRST</b> preliminary amendment. <input type="checkbox"/> A <b>SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.			
14.	<input type="checkbox"/> A substitute specification.			
15.	<input type="checkbox"/> A change of power of attorney and/or address letter.			
16.	<input checked="" type="checkbox"/> Other items or information: <input checked="" type="checkbox"/> PCT Request form <input checked="" type="checkbox"/> Notification of the International Application Number and of the International Filing Date <input checked="" type="checkbox"/> Notification Concerning Submission of Transmittal of Priority Document <input checked="" type="checkbox"/> Notification of Transmittal of the International Search Report <input checked="" type="checkbox"/> Information Concerning Elected Offices Notified of their Election <input checked="" type="checkbox"/> Notification of Transmittal of the International Preliminary Examination Report <input checked="" type="checkbox"/> International Application Published under the Patent Cooperation Treaty <input checked="" type="checkbox"/> References in Search Report: US 5 695 553; WO 98 09925; US 4 315 957; Patent Abstracts of Japan; US 5 437 722; <input checked="" type="checkbox"/> DATABASE WPI , Week 8635, Durwent Publications			

US

Annex US.II. page 2

PCT Applicant's Guide - Volume II - National Chapter - US

534 Rec'd PCT/PTC 07 JUL 2000

U.S. APPLICATION NO. (If known, see 37 CFR 1.51) <b>09/600011</b>	INTERNATIONAL APPLICATION NO. <b>PCT/US99/00064</b>	ATTORNEY'S DOCKET NUMBER <b>P1998J130</b>																
17. The following fees are submitted:		<b>CALCULATIONS</b>																
<b>Basic National Fee (37 CFR 1.492(a)(1)-(5)):</b> Search Report has been prepared by the EPO or JPO ..... International preliminary examination fee paid to USPTO (37 CFR 1.482) ..... No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) ..... Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) ....		PTO USE ONLY																
<b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>		\$ 840.00																
Surcharge of \$130.00 for furnishing the oath or declaration later than Months from the earliest claimed priority date (37 CFR 1.492(e)).		\$ 130.00																
<table border="1"> <thead> <tr> <th>Claims</th> <th>Number Filed</th> <th>Number Extra</th> <th>Rate</th> </tr> </thead> <tbody> <tr> <td>Total Claims</td> <td>22 - 20 =</td> <td>2</td> <td>x \$18.00</td> </tr> <tr> <td>Independent Claims</td> <td>3 - 3 =</td> <td>0</td> <td>x \$78.00</td> </tr> <tr> <td>Multiple dependent claim(s) (if applicable)</td> <td></td> <td></td> <td>+\$260.00</td> </tr> </tbody> </table>		Claims	Number Filed	Number Extra	Rate	Total Claims	22 - 20 =	2	x \$18.00	Independent Claims	3 - 3 =	0	x \$78.00	Multiple dependent claim(s) (if applicable)			+\$260.00	\$ 1,266.00
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a. <input type="checkbox"/> A check in the amount of \$ _____ to cover the above fees is enclosed.																		
b. <input checked="" type="checkbox"/> Please charge my Deposit Account No. <u>05-1330</u> in the amount of \$ <u>1,266.00</u> to cover the above fees. A duplicate copy of this sheet is enclosed.																		
c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>05-1330</u> . A duplicate copy of this sheet is enclosed.																		
<b>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</b>																		
<b>SEND ALL CORRESPONDENCE TO:</b> <u>EXXONMOBIL RESEARCH AND ENGINEERING COMPANY</u> <u>P.O. Box 900</u> <u>ANNANDALE, NEW JERSEY 08801-0900</u>																		
 <b>SIGNATURE</b> <b>Joseph J. Allocca</b>																		
<b>NAME</b> <u>Joseph J. Allocca</u>																		
<input checked="" type="checkbox"/> Pursuant to 37 CFR 1.34(a)																		
<b>REGISTRATION NUMBER</b> 27,766																		

2/PRTS

<sup>1</sup> 534 Rec'd PCT/PTC 07 JUL 2000GYPSUM PRODUCT

This invention relates to a gypsum product and to a process for its manufacture. More particularly, this invention relates to a foamed gypsum product of improved 5 water resistance and/or reduced density and to a process, preferably to a continuous process, for its manufacture.

Gypsum board (or plaster board or wallboard) is used extensively in the construction industry. It typically comprises a substantially flat core of set gypsum on either 10 side of which a liner may be adhered. A liner typically comprises paper. The core may be reinforced; for example, reinforced with glass fibres.

Gypsum products (or Plaster of Paris or plaster products) are produced by mixing anhydrous calcium sulphate 15 or calcium sulphate hemihydrate with water, and permitting the mixture to set thereby producing calcium sulphate dihydrate. Often the slurry is foamed by incorporating a preformed solution of foaming agent in water (a surface active material) before adding to the mould means. A 20 pervasive problem with gypsum products, however, is that calcium sulphate dihydrate absorbs water and this reduces the strength of the gypsum product. Because of this, plaster board (for example) is required, at least in uses where a relatively high humidity is anticipated (for 25 example, kitchens or bathrooms) to be substantially moisture resistant and this requires the presence of a hydrophobing agent. ("Hydrophobing" is a term used in the art to denote a method of preventing, or reducing water absorption).

30 Silicone oil has previously been used as a hydrophobing agent for gypsum products. It is, however, expensive and in relatively short supply. It also has

difficulty in providing a moisture resistance of less than 5 wt % water absorption in the test hereinafter mentioned.

In US-A-5437722 an aqueous emulsion comprising a hydrocarbon wax, a montan wax and emulsifier/stabiliser system and also including a polyvinyl alcohol, is used to render gypsum products water resistant. The emulsifier system may include non-ionic or anionic surfactant and alkali. Examples of non ionic surfactants are alkyl-phenoxy poly(ethyleneoxy) ethanols, sorbitan fatty acid esters and polyoxyethylene sorbitan fatty acid esters. Examples of anionic surfactants are saponified fatty acids.

In our copending application number PCT/GB97/02366, unpublished at the priority date of the present invention, we describe a foamed gypsum product which is hydrophobed by incorporation of an aqueous emulsion comprising a hydrocarbon wax, a montan wax and a colloid stabilised emulsifier system. The preferred colloid stabilised emulsifier system comprises either organic or, more preferably, inorganic colloidal materials. One example is a montmorillonite clay based system in combination with a sodium naphthalene sulphonate.

In the above mentioned PCT application, a comparative example uses an emulsifier system based on a combination of a nonionic surfactant, which was not specifically defined, with an anionic surfactant again, not specifically defined. The emulsifier system used in that comparative example was in fact a combination of an alkyl phenyl ethoxylate with a soap-type anionic surfactant. The worked example using that emulsifier system did not work. In fact it is now believed that the anionic surfactant caused collapse of the foam or that, upon addition to a slurry formed using

relatively hard water, the anionic surfactant was precipitated out of the system by the hardness ions.

The above mentioned PCT application also discloses a comparative example using a nonionic surfactant based 5 emulsifier system including no anionic emulsifier. Whilst this gave some improvement over the mixed system, it was found that using a high enough level of emulsion in the gypsum product to achieve adequate density and/or water adsorption figures lead to over-wetting of paper used for 10 the gypsum product and delamination during production.

In the present invention there is a provided a process for producing a foamed gypsum product involving the following steps:

- 15 a) a slurry of gypsum is formed in water
- b) the slurry is introduced to mould means and allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an 20 emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

- 25 i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point (in saline per DIN 53917) of at least 50; and
- ii) an anionic dispersing agent which is a sulphated compound.

Preferably the anionic dispersant is a so called acid 30 stable compound, that is the compound is ionised over a wide range of pH's including acidic pH. The acid stability can be judged by the  $pK_a$  of the conjugate acid, which should preferably be less than 4, more preferably less than

3, for instance 2 or less. The anionic dispersant is generally a sulphate or a sulphonate.

A suitable class of anionic dispersants are sulphated naphthalene/formaldehyde condensates, for instance having 5 molecular weight in the range 6000 to 40000. These compounds are also known as naphthalene sulphonates. Other aryl sulphonates may also be used. The anionic dispersant is generally used in the emulsion in the form of its sodium 10 salt. Alternatively potassium, ammonium, or even divalent metal salts such as calcium or magnesium, may be used. Suitable compounds are available from BASF AG under the trade name Tamol (trademark).

The nonionic surfactant must be relatively water soluble. The water solubility of non ionic surfactants can 15 be determined by standard test method DIN 53917 in saline. The component should have a cloud point of at least 50, for instance more than 60, up to around 100, for instance approximately 75.

We have found that the nonionic surfactant giving 20 optimum performance is one which has a high foaming ability. Foaming ability can be measured by standard test methods DIN 53902. For instance the test should be carried out according to the method given in sheet 1 of that 25 standard test method, at 40°C, with the surfactant being used in a concentration of 2 g/l in water containing 1.8 mmol Ca ions/l, the duration of the test being 30 seconds. The foaming ability should be at least 300, more preferably at least 500, for instance up to 750. Nonionic surfactants with foaming ability using the above mentioned test method 30 of around 600 are available.

The nonionic surfactant is generally an ethoxylated higher alkyl, alkenyl, alkanoyl or alkenoyl compound.

Alternatively ethoxylated aryl compounds may be used, for instance ethoxylated alkyl phenol derivatives. Preferably the compound is a  $C_{8-18}$ -alkanol ethoxylated with 3 to 30 equivalents of ethylene oxide, for instance a  $C_{12-16}$ -alkanol ethoxylated with 10 to 20 equivalents of ethylene oxide. Suitable compounds are available under the trade name Lutensol (trademark) from BASF AG. It may alternatively be possible to use polyglycosylated alkyl, alkenyl, alkanoyl, alkenoyl and aryl compounds for instance alkyl polyglucosides.

The use of an acid stable anionic dispersant, it is believed, avoids the addition of the emulsion resulting in the collapse of the foam in the gypsum slurry. Accordingly the density of the product is optimised. The use of the anionic dispersant in combination with non ionic surfactant avoids the use of such high concentrations of nonionic dispersant in the gypsum slurry which can lead to over-wetting of paper used in the gypsum product and delamination during production. The preferred surfactant makes the emulsion suitable for use with gypsum slurries made up in hard water, for instance water having hardness value of at least 100 ppm  $Ca^{2+}$  even more than 150 ppm  $Ca^{2+}$ , for instance at least 200 ppm  $Ca^{2+}$ .

The use of the high foaming non ionic surfactant is believed also to contribute to optimisation of the gypsum slurry foam and the density of the final product. The preferred combination of surfactants in the emulsifier system allows high levels of wax to be incorporated into the final product for optimum hydrophobing of the gypsum product.

The emulsifier system has process advantages also in production of non-foamed products, for instance, other

products made on continuous lines such as fire resistance board. The system provides good compatibility with the equipment

The petroleum-derived hydrocarbon wax (a) is preferably one with a high melting point and a low oil content. A preferred such wax is a paraffin wax, such as fully refined paraffin wax. Fully refined paraffin waxes are generally obtained from highly paraffinic refinery streams such as those obtained from the solvent dewaxing of distillates and other lube fractions. The product is further typically characterised as follows:

CHARACTERISTICS	TEST METHOD	SPECIFICATION	
		MIN	MAX
Congealing Point (°C)	ASTM D938	55	69
Oil in Wax (%)	ASTM D721		1
Penetration at 25°C (mm/10)	ASTM D1321	10	20
Penetration at 50°C (mm/10)	ASTM D1321		80
Viscosity (cSt @ 100°C)	ASTM D445	3	7

An example of a fully refined paraffin wax which has been found to be entirely satisfactory, and which satisfies the above specification, is MOBILWAX 135 (derived from the 150 SPN stream) as supplied by Mobil Oil Company Limited; MOBILWAX 145 or 150 (derived from the 300 or 450 SPN stream) are also suitable. While these waxes are hydrofinished to give a white colour and good odour, unfinished wax (which differs only in colour and odour) is also suitable for use in accordance with the invention.

The petroleum-derived hydrocarbon wax a) suitably comprises from 20 to 40 wt % of the aqueous emulsion, preferably from 25 to 35 wt % of the aqueous emulsion.

The montan wax or lignite wax b) is another wax with a high melting point. It is preferably used in crude (or raw) form. Such a product is typically characterised as follows:

	CHARACTERISTICS	TEST METHOD	SPECIFICATION		PREFERRED SPECIFICATION	
			MIN	MAX	MIN	MAX
10	Congealing Point (°C)	ASTM D938	67	80	75	80
15	Acid Value (mg KOH/g)	ASTM D1980	10	37	10	20
20	Saponification Value (mgKOH/g)	ASTM D1962	35	100	65	90
25	Ash Content (% wt)	ASTM D482		1		1.0
	Density at 20°C (g/cm³)	ASTM D1298	0.95	1.04	0.95	1.04
	Viscosity (cSt at 90°C)	ASTM D445	20	400	150	400
	Viscosity (cSt at 100°C)	ASTM D445	20	200	60	150

The montan wax b) suitably comprises from 10 to 20 wt % of the aqueous emulsion, preferably from 11 to 15 wt % of the aqueous emulsion.

The emulsifier system (i.e. the total of two or more components of a multi-component system) is suitably present in an amount from 0.5 to 6 wt %, preferably 12 to 5 wt %, more preferably 1.5 to 4% of the aqueous emulsion. The

ratio of the non-ionic and anionic components is preferably in the range 5:1 to 1:5, more preferably 3:1 to 1:3, most preferably 2:1 to 1:2.

In the invention the slurry in water, preferably 5 contains 100 parts by weight of gypsum and from 0.5 to 10, preferably from 1 to 5 % by weight of an emulsion as herein defined. The slurry suitably contains 50-60 weight % gypsum and 40-50 weight % water, preferably about 55% gypsum. An accelerator is usually added, for instance a 10 slurry mix from a previous batch.

The slurry preferably contains a foaming agent. Preferably the product is a paper lined board and the process thus preferably includes a step of foaming, usually involving formation of a pre-formed foam by vigorous 15 stirring of the foaming agent in water, followed by mixing the prefoam into preformed gypsum slurry containing the emulsion.

This invention also provides a water-resistant gypsum product which comprises the set composition. Whilst the 20 product may be an unlined board, the invention is particularly applicable to a product which comprises a core product of a set such composition sandwiched between a pair of liners usually paper liners. Another suitable product is fire resistant board which has a glass fibre scrim 25 embedded in each surface of the gypsum board, which is generally unfoamed. The invention includes also the emulsion itself and the process for making it.

The invention further provides a process for the preparation of a water-resistant gypsum board product, 30 which process comprises forming a mixture which is a slurry in water containing 100 parts by weight of gypsum and from 0.5 to 5 parts by weight of an emulsion according to the

invention; forming a layer of the mixture in a mould means and drying the layer of gypsum mixture, while permitting hydration of the gypsum, for form a board product. Preferably the process is continuous. The process may be 5 for forming tiles or blocks or boards. Blocks may be formed in moulds from which they are removed when set. Tiles or boards may be formed by spreading a layer of the gypsum mixture on a first planar substrate, a second planar substrate is positioned over the layer to form an assembly, 10 and the mixture is allowed to set in the assembly. A gasket may be provided between the planar substrates. Where the product is a lined board, the first and second planar substrates are constituted by liner, for instance paper, usually supported in a mould. Where the product is 15 to be unlined, the planar substrates are removed when the product is set. Where the product is a block, it is usually removed from a mould before the mixture is completely set, but when it is hard enough to handle. Where the product is a fire resistant board a fibreglass 20 scrim is embedded in each side of the slurry in the mould means by feeding to each side of the poured (unfoamed) slurry before the nip forming a dam in an apparatus similar to that shown in Figures 3 and 4 below.

This invention includes the use of an aqueous emulsion 25 of the invention to furnish a gypsum product with water resistance and the use of an aqueous emulsion of the invention to aid foaming of a gypsum slurry, for instance to reduce the density of the set foamed gypsum product.

Figure 1 shows the water absorption results of the 30 product of the comparative example.

Figure 2 shows the water absorption results of the product of the example of the invention.

Figures 3 and 4 are a schematic representations of a foaming station for lined gypsum board.

The following Example illustrate the invention.

EXAMPLE

5       First the wax phase was prepared by dissolving 12 wt % crude montan wax (Crude Montan Wax supplied by Schuemann Sabol GmbH) in 30 wt % of fully refined paraffin wax (MOBILWAX 135 supplied by Mobil Oil Company Limited) at a suitable raised temperature. 1% by weight sodium 10 naphthalene sulphonate (a sulphated naphthalene formaldehyde condensate having a molecular weight of about 6000 to 40000) from the TAMOL (trademark) range supplied by BASF was added to water to form the aqueous phase and stirred for a period. 0.45% Non ionic surfactant (added as 15 a 90% aqueous solution) (a C<sub>13</sub> alkanol - 12 mole ethoxylate available as Lutensol TO12 series) was then added to the aqueous phase and stirred for a period. The temperature of the wax melt was lowered to 100°C and the wax phase was next added to the aqueous phase heated to a suitable 20 temperature, with stirring for a suitable period to form a pre-emulsion. The pre-emulsion still at a raised temperature was next recycled through a homogeniser, with no impressed pressure, for a full pass. Gradually, the pressure was increased to a value in the range 20-25 MPa 25 (220 bar) and the emulsion recycled for a further pass to form an aqueous emulsion in accordance with the invention.

The emulsion was then tested for its performance in the production of a gypsum product. A conventional foaming agent was mixed with vigorous stirring with a suitable 30 quantity water to generate a foam mixture. A gypsum slurry mix was prepared by adding a predetermined amount (1.4, 1.6 or 1.8% by weight based on the amount of gypsum) of wax

emulsion (according to the invention or comparative) to around 40 parts by weight water along with predetermined amounts of a wetting agent, starch and an accelerator in a total amount of 0.38 parts by weight. To this around 58 parts by weight gypsum was added with stirring. The pre-generated foam mix was next added to the gypsum slurry and stirred to form a foamed gypsum slurry. The slurry was poured into a paper lined mould of 300 x 300 x 12.5 mm dimension and a second sheet of paper placed on top to form a gypsum coupon which was then dried in three stages of successively lower temperatures and longer times to a constant weight. The density and 2 hour water absorption were then determined. The density was calculated by dividing the dry weight of the test specimen by the mould volume. The water absorption was determined by cutting a test specimen measuring 280 x 280 mm from the coupon and immersing this specimen in a water bath at 23°C covered with 25 to 35 mm of water for 2 hours. Its weight before and after immersion was measured and the percentage increase calculated.

The results, which include comparative tests, are shown in figures 1 and 2. In these tests density and 2 hr water absorption were measured and reported using a the emulsifier system of the invention and also as comparison, based on the colloid emulsified system of the above mentioned PCT publication, in which the same amounts of wax emulsion containing an emulsifier system of a bentonite clay and sodium naphthalene sulphonate.

A further example has also been conducted in which the emulsion containing Mobilwax 135, which has a melting point (congealing point) in the range 57-60°C and a maximum content of oil of 1.0wt%, is used at a level of 4% by

weight. This example is then repeated, but using emulsions (at 4% by weight in the gypsum) in which the Mobil wax 135 is replaced by waxes having higher (63-66°C, and 66-69°C, respectively) and lower (54-57°C) melting/congealing points. When used at the same levels in the emulsion, the emulsion (at the same level in the gypsum) produced good results for water absorption. The values were less than 5%, indeed less than 2.5%, in each case.

In Figures 3 and 4 there is shown a forming station for gypsum board manufacture is shown generally at 100. It comprises a conveyor 1 which is formed from an array of like, generally coplanar, parallel driven rollers 2 which are rotatable in the same sense. Above the conveyor is a manifold mixing box 3 into which entry conduits 4 and 5 and a plurality of exit nozzles 6,6',6" are flowably connected. A contra-rotatable roller 7 is biased toward rollers 2 to form a nip 8.

In use, a plaster slurry 9 and the pre-generated foam mix 10, detailed above, are supplied under gravity, in an appropriate ratio, through conduits 4 and 5, respectively, into mixing box 3 where they are mixed to form a foamed plaster mixture 11. The foamed plaster mixture is then sprayed through the plurality of nozzles 6,6',6" onto a lower paper liner 12 which is being continuously conveyed, in the direction indicated, by rollers 2. The foamed plaster mixture becomes substantially evenly distributed across, and adheres to, the paper liner. An upper paper liner 13 is continuously conveyed, in the direction indicated by roller 7 into nip 8 where buildup of, the foamed plaster mixture into a dam 9 occurs and the plaster mixture adheres to the paper 13. Uncured plasterboard 14 is continuously conveyed downstream from the forming

station along a long conveyor belt allowing the chemical reactions of setting to take place. It is then cut to the required length; and dried by passage through multideck drying zones.

5        In an alternative process, which is not illustrated, a non-foamed slurry is poured into block shaped moulds, in which it is allowed to set partially. The blocks are hard enough to handle after a few minutes, at which time they are pushed from the mould using, for instance, a hydraulic jack, and are conveyed to an oven to complete the setting/drying process.

10

CLAIMS

1. A process for producing a gypsum product involving the following steps:
  - a) a slurry of gypsum is formed in water
  - 5 b) the slurry is introduced to mould means and the gypsum allowed to hydrate,  
in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system is added to the slurry before introduction into the mould means, and is characterised in  
10 that the emulsifier system comprises:
    - i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at  
15 least 50; and
    - ii) an anionic dispersant which is a sulphated compound.
2. A process according to claim 1 in which the process is continuous and involves pouring of the slurry  
20 onto a continuously moving belt.
3. A process according to claim 1 or 2 in which the gypsum slurry is foamed before introduction into the mould means.
4. A process according to claim 1 in which the  
25 anionic dispersant is a sulphate or a sulphonate.
5. A process according to claim 4 in which the anionic dispersant is a polymeric compound, preferably an aryl sulphonate.
6. A process according to claim 5 in which the  
30 anionic dispersant is a naphthalene sulphonate, preferably the sodium salt.

7. A process according to any preceding claim in which the nonionic surfactant is a higher alkanol, alkenol, alkanoic or alkenoic acid or aryl alcohol (including phenol) or carboxylic acid ethoxylated with at least 2 equivalents of ethylene oxide, preferably up to 100, for instance 3 to 30 equivalents, ethylene oxide.

8. A process according to claim 7 in which the nonionic surfactant is a  $C_{8-18}$ -alkanol or -alkenol ethoxylated with 3 to 30 moles of ethylene oxide.

9. A process according to any preceding claim in which the petroleum-derived hydrocarbon wax (a) is one with a high melting point and a low oil content, preferably a paraffin wax, more preferably such a wax having a congealing point in the range 55 to 69°C (ASTM D938) a penetration value (by ASTM D1321) at 25°C in the range 10 to 20 mm/10 and at 50°C at least 50 mm/10 and a viscosity at 100°C (by ASTM D445) in the range 3-7 cst.

10. A process according to any preceding claim in which the montan wax has a congealing point in the range 67-80°C, an acid value (by ASTM D1980) in the range 10 to 37 mgKOH/g, a saponification value (by ASTM D1962) in the range 35 to 100 mgKOH/g, a viscosity (by ASTM D445) at 90°C in the range 20-400 cst and at 100°C in the range 20 to 200 cst.

11. A process according to any preceding claim in which, in the emulsifier, the montan wax is present in an amount in the range 10 to 20% by weight, the hydrocarbon wax is present in an amount in the range 20 to 40% by weight and the emulsifier system is present in an amount in the range 1 to 6% by weight.

12. A process according to any preceding claim in which the ratio of the anionic dispersant to nonionic

surfactant in the emulsion is in the range 5:1 to 1:5, preferably 3:1 to 1:3.

13. A process according to any preceding claim in which the emulsion is added to the gypsum slurry in an 5 amount in the range 0.5 to 10%, preferably in the range 1.0 to 5.0% by weight based on the weight of gypsum.

14. A process according to any preceding claim in which the mould means has a paper liner which becomes permanently laminated to the solidified gypsum.

10 15. A process according to any preceding claim in which the water in which the gypsum slurry is formed has a hardness of at least 100 ppm  $\text{Ca}^{2+}$ , preferably at least 150 ppm  $\text{Ca}^{2+}$ , more preferably at least 200 ppm  $\text{Ca}^{2+}$ .

15 16. An emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system **characterised in that** the emulsifier system comprises:

- 20 i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- ii) an anionic dispersant which is a sulphated compound.

17. An emulsion according to claim 16 having the further features defined in any of claims 2 to 12.

25 18. A method of forming an emulsion in which a petroleum derived hydrocarbon wax and a montan wax are each melted and blended in molten form, an emulsifier system is dissolved into water to form an aqueous emulsifier solution and the molten wax mixture is dispersed into the aqueous emulsifier solution to form an emulsion, **characterised in** 30 **that the emulsifier system comprises:**

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- 5 ii) an anionic dispersant which is a sulphated compound.

19. A method according to claim 18 in which the emulsifier system is as defined in any of claims 2 to 8 and 12 and/or the waxes are as defined in claim 9 and/or claim 10.

10 20. A method according to claim 18 or 19 in which the montan wax is used in an amount in the range 10 to 20% by weight of the emulsion, the hydrocarbon wax is used in an amount in the range 20 to 40% by weight of the emulsion and the emulsifier system is used in an amount in the range 0.5  
15 to 6% by weight, preferably 1 to 2.5% by weight of the emulsion.

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## ABSTRACT

## GYPSUM PRODUCT

A wax emulsion comprising an emulsifying system containing a sulphated anionic surfactant and a non ionic surfactant having high water solubility (cloud point) and high foaming ability is added to a gypsum slurry to improve the moisture resistance of gypsum board. The wax is a mixture of a petroleum derived hydrocarbon wax and a montan wax.

1/2

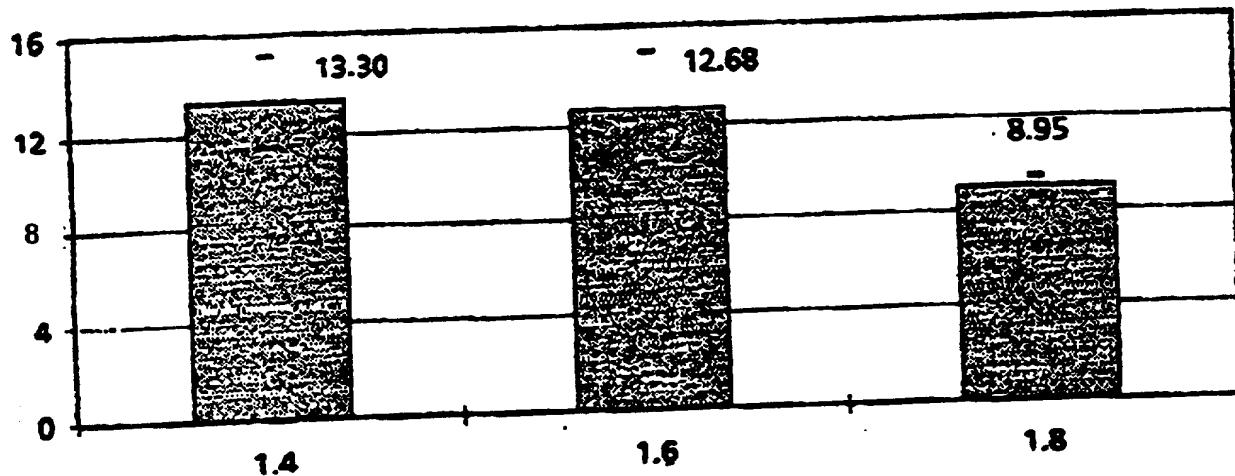


Figure 1

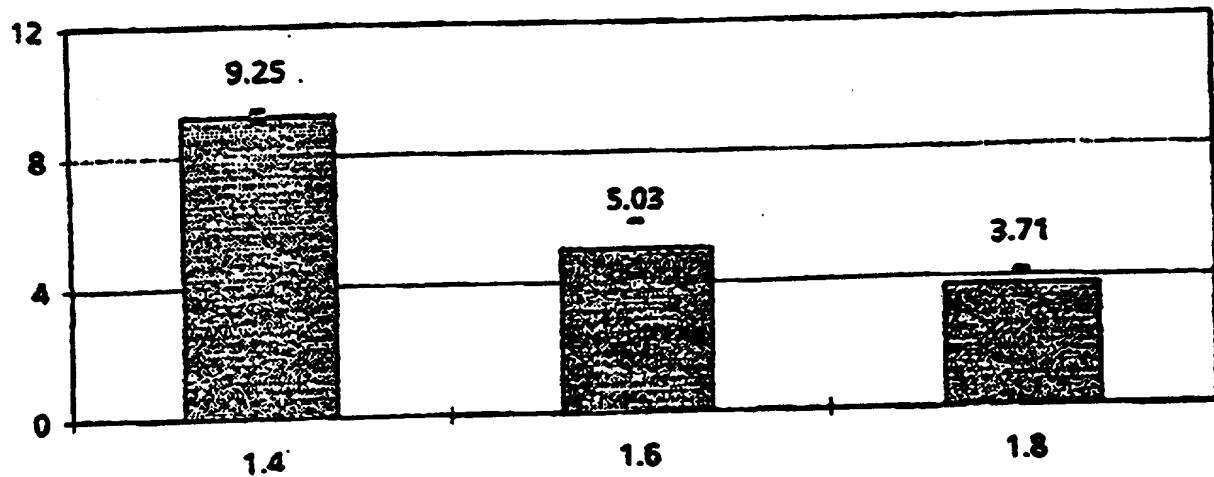


Figure 2

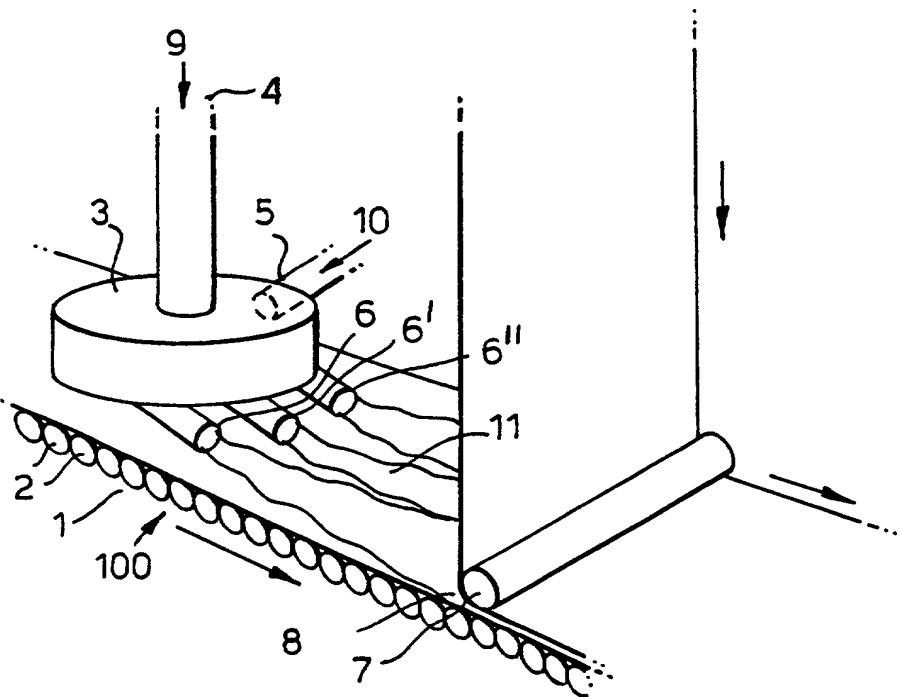


Figure 3

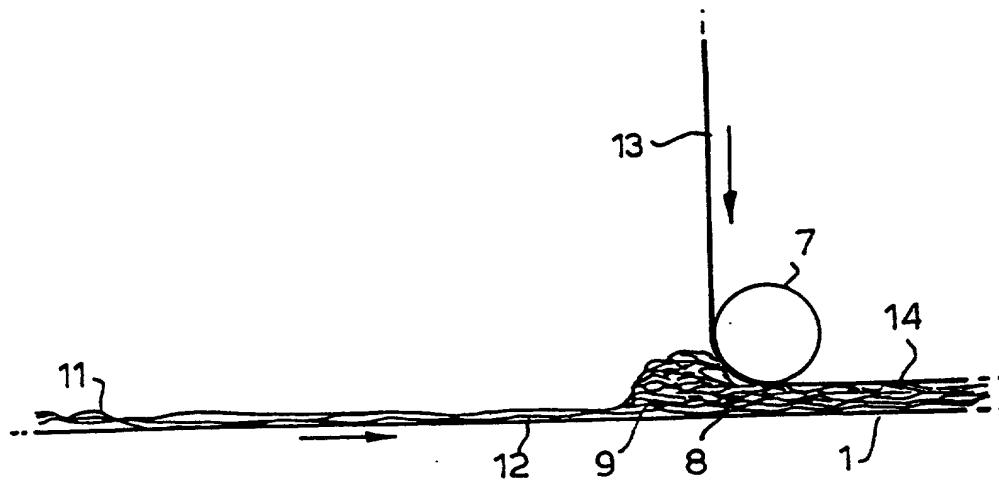


Figure 4

USA

DECLARATION AND POWER OF ATTORNEY

As a below-named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name; I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of subject matter which is claimed and for which a patent is sought on an invention entitled  
**GYPSUM PRODUCT**

the specification of which  is attached hereto or

was filed on 08 JAN 1999 as United States Application Number or PCT International Application Number PCT/GB99/00064 and was amended on (if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56. I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for a patent or inventor's certificate, or PCT international application having a filing date before that of the application on which priority is claimed:

Prior Foreign Application Number(s)	Country	Foreign Filing Date	Priority Not Claimed	Certified Copy Attached?
			YES	NO
9800368.4	GB	08 JAN 1998	<input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/>

As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

Direct all correspondence to:

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C 1001 and that such willful false statements may jeopardise the validity of the application or any patent issued thereon.

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